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(54) Title: CHEMICAL COMPOSITION

(57) Abstract: A water dispersible granular composition containing an agrochemical active agent, particularly a low use-rate active, and a particular basic, inorganic salt wherein a 1% aqueous solution of the composition has a pH of at least 8.0, and which is made by extruding a premix in the form of a free-flowing powder and without forming a paste provides improved bioavailability of the active. The invention is particularly useful in improving delivery of active from compositions having mixed actives and low levels of sulphonamide active.

WO 2005/120227 A2

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## CHEMICAL COMPOSITION

The invention relates to a novel agrochemical chemical composition and its use. The invention is more particularly concerned with a composition containing an agrochemical agent and a basic inorganic salt which delivers the active compound efficiently to a substrate which is to be treated.

Compositions containing a biologically active compound typically require the active to be delivered to a substrate to be treated, for example a crop. Such delivery often occurs in an aqueous environment, for example, by dispersing and optionally dissolving the composition in which the active is formulated in water and applying the solution or dispersion to the substrate. In delivering the active, the composition will typically disperse in aqueous medium and begin to dissolve if it is soluble, hence the rate and degree of dispersion and dissolution may effect the overall delivery of the active to the substrate to be treated. The active may also be applied in solid form to or near to the substrate and then be mixed with aqueous solution. Thus, a solid agrochemical composition may be applied to a plant or to a locus to be treated and water then added to the agrochemical to dissolve and/or disperse the agrochemical *in situ*.

Where dissolution or dispersion of a composition is incomplete, the full dosage of active agent in the composition may not be delivered to its target substrate. Accordingly, the dosage of active to be delivered may be lower than that intended and may introduce uncertainty as to the actual dose of active that reaches the substrate in a bioavailable manner. Use of a higher dose to achieve a certain effect may therefore be required but uncertainty in the actual dosage delivered remains, product usage may be inefficient, may have a deleterious effect and be of greater cost, the active agent typically being the most expensive component in a composition. Further, uncertainty in the dosage reaching the desired target substrate may leave residual, undelivered active agent in the vicinity of its application so potentially leading to environmental damage or undesirable localised concentration..

Once formulated, an agrochemical composition should also suitably retain adequate chemical and physical stability over a reasonable time so enabling the product to be stored and distributed without degradation to a material extent.

WO 01/13721 discloses a water dispersible agrochemical composition comprising a low use rate agrochemical active which is present at a level of less than 50% by weight of the composition and a dispersing agent, suitably a non-ionic or anionic surfactant. The compositions of WO 01/13721 exhibit good delivery characteristics as compared to earlier known compositions and provide adequate stability.

However, providing further improved delivery, and hence bioavailability, of an active agent without compromising the stability of the product to reduce or avoid the aforementioned disadvantages, especially where the composition or active agent dissolves or disperses poorly in an aqueous medium, remains desirable. In addition to the components of the product, the process by which they are combined may have a significant effect on the delivery of the active.

We have now found that improved delivery characteristics of an agrochemical composition, particularly a water dispersible granule produced in an extrusion process, may be secured by including a basic, inorganic salt in combination with an agrochemical active such that upon dissolution the composition provides a basic solution and by avoiding formation of a paste prior to extrusion of the components to form granules. We have also found that such granules exhibit acceptable stability. We have further found that improved delivery is found particularly for compositions containing a low level of agrochemical active or combination of actives and a higher proportion of the active than previously known may be delivered in a short period of time after dissolution

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Accordingly, a first aspect of the invention provides a process for the production of a granular composition comprising an agrochemical active agent and a basic, inorganic salt and optionally a dispersing agent wherein a 1% aqueous solution of the composition has a pH of at least 8.0 when subjected to CIPAC Test MT75.3 which process comprises forming a premix in the form of a free-flowing powder comprising the active, the basic inorganic salt and optionally a dispersing agent with at least one of the components of the pre-mix being liquid, without forming a paste, extruding the pre-mix to form the granules and optionally drying the granules.

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The invention is especially beneficial in enhancing the delivery of the agrochemical active in a water dispersible granular composition.

5 The invention provides, in a second aspect, a method of increasing the rate and degree of dissolution of an agrochemical active in an aqueous medium which method comprises formulating a composition comprising the agrochemical active with a basic, inorganic salt such that a 1% aqueous solution of the composition has a pH of at least 8 when subjected to CIPAC Test MT75.3 and suitably producing the composition by extruding a premix comprising the active and the salt and optionally a  
10 dispersing agent wherein a paste is not formed prior to extrusion.

The composition in the second aspect of the invention is suitably produced by a process comprising forming a premix in the form of a free-flowing powder comprising the active, the basic inorganic salt and optionally a dispersing agent with at least one  
15 of the components of the pre-mix being liquid, without forming a paste, extruding the pre-mix to form the granules and optionally drying the granules.

The invention provides in a third aspect use of a basic, inorganic salt in an agrochemical composition, said composition comprising an agrochemical active and  
20 the said salt, and suitably being produced by an extrusion process without the formation of a paste prior to extrusion, to increase the level of solubility of the active when mixed with aqueous solution and thereby to improve delivery of the active to a plant or a locus to be treated.

25 The increase in rate and degree of dissolution of a composition as described in the second aspect of the invention or the level of solubility of the active described in the third aspect of the invention is to be compared to the rate and degree of dissolution under the same conditions of a comparative composition having the same components at the same level as the composition of the invention and made by the  
30 same process except that the comparative composition comprises an inert filler typically used in agrochemical formulations, for example starch, lactose, china clay and kaolin, in place of the inorganic basic salt and provided that the filler is such that a 1% aqueous solution of the comparative composition has a pH of less than 8 when subjected to CIPAC Test MT75.3

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By incorporating a basic, inorganic salt in the composition such that a 1% solution of the composition provides a pH of at least 8, preferably at least 8.5 and especially at least 9, and avoiding the formation of a paste prior to extrusion, the rate and degree of solubility of the active in aqueous solution may be increased so providing excellent bioavailability of the active.

The invention is particularly applicable to a low use rate agrochemical actives as they may often exhibit poor solubility or dispersibility in water under conditions of use for example bensulfuron-methyl. As employed herein, the term "low use rate" active denotes those agrochemical actives which typically are applied at a rate of less than 100g/hectare and the term "high use rate" active denotes those agrochemical actives which typically are applied at a rate of more than 1000g/hectare.

Suitable low use-rate agrochemical actives include abamectin, azoxystrobin, bensulfuron-methyl, carfentrazone-ethyl, chlorsulfuron, cinosulfuron, clodinafop, clopyralid, lambda-cyhalothrin, deltamethrin, diflufenican, emamectin benzoate, fibronil, flurtamone, imazamethabenz-methyl, imazapyr, imazethapyr, imadacloprid, metsulfuron-methyl, milbectin, nicosulfuron, pirimisulfuron-methyl, rimsulfuron, sulfometuron-methyl, thifensulfuron-methyl, tribenuron-methyl, and triflurosulfuron-methyl. Preferably the low use rate active comprises a sulfonyl urea, for example bensulfuron-methyl and chlorsulfuron.

The level of the agrochemical active is suitably selected according to the particular compound to be used and the intended application. In the case of a low use rate active, a concentrated composition may be employed for high levels of application for example at least 60g/ha, and the composition suitably contains at least 10%, preferably at least 50% active and especially 50 to 70% active. Where lower levels of application are required, for example less than 50g/ha, the composition suitably contains less than 10% active, preferably less than 7 % active and especially 1 to 7% active. Suitably, the composition will contain the active at a level at which, on mixing with a liquid carrier, for example water, provides a concentration of active which will provide a beneficial effect in treating the plant or locus to be treated.

The composition produced according to the invention dissolves rapidly in an aqueous environment to form a solution of the active material which has a higher proportion of the active in solution than would a composition having the same level of active but with an inert filler in place of the basic, inorganic salt. This characteristic enhances the efficacy of the active agent by improving delivery of it to the plant or locus to be treated. This enhanced bioavailability means that the effect of the reactive agent may be obtained using a lower total amount of the active agent as desired. In the case of an agrochemical, this provides environmental benefits through lower use rates to achieve a given effect.

A practical advantage afforded by the invention is that a lower level of active may be contained in the composition yet, as a higher proportion dissolves, still provide delivery of a given dose of the active. Furthermore, with a lower level of the active in the composition than may be conventional to achieve a given delivered dose of the active, there is a reduced risk that some of the active will not be dissolved in the liquid carrier. Accordingly, the risk of high localised concentrations of the active due to poor dissolution in an aqueous environment may be reduced. Penetration to the crop, especially the roots of plants, possibly leading to crop damage, of high localised concentration of active may be reduced.

A further advantage is that a lower level of active ingredient provides enhanced flexibility in formulating a composition as compared to conventional compositions employing high levels of active. Thus, the formulator may include a secondary active ingredient or other materials as desired in the composition to provide an optimum effect or balance of properties.

The basic, inorganic salt is suitably an alkali metal salt, for example a sodium salt and a potassium salt. The anion of the salt may be any anion provided that when the salt is formulated with the selected active to provide a biologically active composition, a 1% aqueous solution of the composition has a pH of at least 8 when tested according to CIPAC Test MT75.3. In a preferred embodiment, the basic inorganic salt comprises an alkali metal carbonate, phosphate, hydrogen carbonate, hydrogen phosphate, trimetaphosphate, hexametaphosphate, polyphosphate, tripolyphosphate and pyrophosphate. Potassium and sodium carbonate are especially preferred. The basic inorganic salt may be milled to a desired particle size, for example 1 to 20

microns as desired and suitably imparts improved solubility characteristics to the composition.

Suitably the basic inorganic salt is present at a level of at least 1%, preferably at least 3% desirably, the salt is present at a level that does not exceed 40% and preferably does not exceed 30%.

It has been found that use of an anhydrous salt in the composition provides excellent solubility characteristics but does not lead to an unacceptable deterioration in chemical or physical stability of the composition. Suitably, the invention comprises an anhydrous, basic, inorganic salt, preferably an anhydrous sodium salt, for example anhydrous sodium carbonate. A basic inorganic salt shall be considered as anhydrous if it contains no water or, if present, does not exceed 2% by weight.

Examples of particularly preferred combinations of active agrochemical and basic inorganic salt include bensulfuron-methyl and sodium carbonate and chlorsulfuron and sodium carbonate. Suitably the sodium carbonate is anhydrous.

For a known composition containing less than 10% active, the amount of active dissolved after 15 minutes at 5°C as a percentage of the amount of active in the composition may typically be less than 65% or even less than 50%.

Suitably, for a composition produced according to the invention containing an active agent at a level up to 10% by weight of the composition, at least 70%, desirably 80% and preferably at least 90% of the active agent in the composition is dissolved in water after 15 minutes at 5°C. Preferably at least 60% or more suitably at least 70% of the active agent is dissolved after 5 minutes. Optimally, the proportion of active dissolved after 15 minutes at 5°C for a composition produced according to the present invention is at least 30% and desirably at least 45% more than the proportion of active dissolved in a comparative composition.

In known agrochemical compositions, the amount of active dissolved after 15 minutes at 5°C as a percentage of the amount of active in the composition may typically be less than 20% or even less than 15% for a composition containing at least 50% active. Suitably, for a composition containing more than 10% and

preferably more than 50% of active agent, suitably at least 25%, desirably at least 40% and preferably at least 75% of the active agent in the composition is dissolved after 15 minutes at 5°C. Preferably at least 40% is dissolved after 5 minutes. Optimally, the proportion of active dissolved after 15 minutes at 5°C for a composition produced according to the present invention is at least 100% and desirably at least 200% more than the proportion of active dissolved in a comparative composition.

The dissolution of a composition may be tested by the method set out in the examples under the heading "Solubility Data".

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Preferably the weight ratio of total agrochemical active in the composition to basic inorganic salt is not more than 10:1 and desirably not more than 6:1. Preferably the ratio of total agrochemical active in the composition to basic inorganic salt is at least 1:3 and desirably at least 1:1. In a preferred embodiment the ratio is from 1 to 12:2 and more preferably 1 to 6:1. Optimally, the total level of the active is not less than that of the basic inorganic salt and is most preferably from 4 to 1:1 for compositions having a total active of less than 10%. For compositions with an total active level greater than 10%, the total level of active to basic inorganic salt is desirably from 4 to 2:1. By way of examples, a composition having a total active level of 5% will, optimally, have an inorganic basic salt at a level of about 1 to 5% and a composition having a total active level of 60% will, optimally have an inorganic basic salt present at a level of 15 to 30% by weight of the composition.

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The compositions produced according to the present invention result in the individual components being used at lower rates and with less phytotoxicity than conventional compositions of the said active materials, against a wide range of pests and diseases.

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In a preferred embodiment, the composition comprises a second active. In a preferred embodiment the second active is a high use rate active. Suitable high use rate actives include atrazine, benomyl bentazone, bifenox, bromoxynil, captan, carbendazim, chloridazon, chlorothalonil, chlortoluron, cyhexatin, cymoxynil, alpha-cypermethrin, deltamethrin, dimethomorph, diuron, ethofumesate, glyphosate, imazamethabenz-methyl, imazapyr, imazethapyr, isoproturon, linuron, mancozeb, maneb, metamitron, methiocarb, metribuzin, oxyfluorfen, phenmedipham, propanil,

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propyzamide, simazine, and thiram. Preferably the high use rate active comprises propanil or isoproturon.

5 Examples of particularly preferred combinations of a high use rate, a low use rate active agrochemical and basic inorganic salt include bensulfuron-methyl, propanil and sodium carbonate and chlorsulfuron, isoproturon and sodium carbonate. Suitably the sodium carbonate is anhydrous.

10 Suitably a high use rate active is present at a level of at least 30%, more preferably at least 50%, optimally at least 65%, for example 75% by weight of the composition.

Suitably the composition comprises a dry-free-flowing, dustless and rapidly dissolving granular formulation.

15 The second active may be added to the premix with the first active or the actives may be added sequentially as desired.

20 The composition suitably comprises a dispersing agent so as to aid the rapid dispersion and dissolution of the active material upon dilution and subsequent application of the composition. Suitably, the weight ratio of dispersing agent to the active ingredient in the composition is 0.1 to 10:1, preferably 0.4 to 6:1, for example about 4:1 and about 5:1.

25 In a preferred embodiment, the dispersing agent comprises a surfactant with non-ionic surfactants and especially anionic surfactants being preferred. Example of suitable dispersing agents include alkali metal, preferably sodium salts of lignosulphonates, naphthalene sulphonate formaldehyde condensates, tristyrylphenol ethoxylate phosphate esters, aliphatic alcohol ethoxylates, alkylphenol ethoxylates, ethylene oxide/propylene oxide (EO-PO) block copolymers, "comb" graft copolymers  
30 and polyvinyl alcohol-vinyl acetate copolymers. Other dispersing agents known in the art may be employed as desired.

Suitably the dispersing agent has a Zeta Potential of about 0mV for a non-ionic surfactant and suitably in excess of -30mV or an anionic surfactant and in excess of  
35 +30mV for a cationic surfactant.

In addition to the dispersing agent, other components may be present in the composition for example a wetting agent. Suitable wetting agents include: alkali metal salts of alkylaryl sulphonates, alkyl aryl sulphosuccinates and alkyl sulphates, preferably as the sodium salt.

Other wetting agents, and other excipients known to those skilled in the art may be employed as desired including disintegrants for example: bentonite, modified starch and polyvinyl pyrrolidone; stabilisers, for example citric acid, polyethylene glycol and butylated hydroxy toluene; and fillers, for example, starch, lactose, china clay, sucrose and kaolin; and flow-aids. Preferably, the composition comprises a filler, for example lactose which is beneficial for solubility and a mix of lactose and china clay which may aid extrusion of the components of the composition to form a granule.

The composition of the invention is preferably prepared by the method described in EP-A-1161145 the contents of which are hereby incorporated by reference. Suitably the process comprises, preparing a mix in the form of a free-flowing powder; preferably a homogeneous powder, comprising the active and the basic inorganic salt and optionally a dispersing agent and other components without forming a paste, and extruding the pre-mix in an extruder, preferably without forming a paste, for example a low pressure extruder to form the granules which may optionally be dried. A pre-mix optionally containing other components, for example a second active ingredient may be mixed with the active and basic inorganic salt to form the mix for extrusion. A liquid component is suitably present and a liquid dispersing agent may be used in which case an additional liquid component is not required although a further liquid component may be included as desired. If water has been added to the premix then this may be removed by drying.

Suitable apparatus for the blending step(s) include a low-shear, high intensity blender such as a Lodige Ploughshare mixer, ribbon, Y-cone, double cone or trough blender, so that a free-flowing powder is formed. The mix is fed directly or indirectly into a suitable low-pressure extruder, such as that described in WO 96/26828, so that the premix is compacted against the apertures in the screen and forced through.

In a preferred embodiment, the composition of the mix and the extruder settings are such that the formation of a paste before extrusion is avoided and the material being processed remains a free flowing particulate material during the formation of the pre-mix and suitably during extrusion. In particular, the material optimally does not form a paste prior to extrusion. However, as the composition may contain one or more liquid components, it may be wet or dry provided that it remains free-flowing and particulate during the process. In this context, a paste may be considered as a mass of material, for example an agglomerate, which contains sufficient liquid or is at such a temperature that the particular material being processed forms into an agglomerate which is mouldable or deformable and which is not free-flowing. Thus, a paste does not disintegrate into finer particles on application of shear, for example by rubbing between fingers, but rather remains as an agglomerated mass and the shear acts to mould or deform the agglomerate.

If desired, the components of the composition, either in sequence, all together or some in sequence and others together are first mixed, for example in a blender so that a uniform blend is obtained which is then passed through a suitable milling system such as an air mill, pin mill or air-swept impact mill so that a fine powder \*(the pre-mix) suitably comprising an average particle size of 0.5 to 20 microns, or more preferably between 0.5 to 5 microns is obtained. The powder thus obtained is suitably agglomerated, so that uniform, dust-free granules are obtained, preferably by the process described in EP-A-1161145. This preferred method involves the extrusion of the wetted powder which is then in the form of a freely flowing homogenous powder, in a low temperature, low pressure extruder, for example as described in EP-A-812256.

Where present, the low use rate and high use rate agrochemicals may be combined in the formation of the dry pre-mix with the other formulation ingredients for example dispersing agents or alternatively the pre-mix may be prepared with one of the agrochemicals and the other added to the milled pre-mix. This alternative approach is preferred when the high use rate active is propanil which is suitably incorporated in the dry pre-mix, and the low use rate active is then added to the pre-mix and blended with it prior to granulation.

In a further aspect, the invention provides a method of treating a plant by applying a herbicidally effective amount of a composition produced according to the present invention to the plant or to the locus of the plant.

- 5 The present invention enables the composition to be used at a lower rate of use, that is at a lower mass of composition per unit area to achieve a given effect than known compositions. Suitably the agrochemical active is applied to the plant or locus of the plant at a rate of use of less than 75%, more preferably less than 50% of the conventional rate of use for the active in commercially available compositions.

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- In a preferred embodiment, a composition comprising a sulfonyl urea low use rate active for example bensulfuron-methyl, is applied in use at a rate of use of less than 50g/hectare, especially less than 30g/hectare and optimally less than 20g/hectare. Typically, a commercially available composition containing in excess of 50% by weight of the composition of bensulfuron-methyl may be employed at a rate of use of 15 60g/hectare or more. In another preferred embodiment, the composition comprises a high use rate secondary active comprising propanil in addition to a sulphonyl urea active, for example bensulfuron, and suitably the secondary active is applied in use at a rate of less than 7000g/hectare, preferably less than 5000g/hectare and 20 especially at a rate of less than 3200g/hectare.

- Where the plant is a weed, suitably, the treatment is such as to control or kill the weed. Generally, the composition is applied to the plant or its locus by means of a liquid carrier, typically water, with which the composition is mixed prior to application. 25 If desired, the composition may be mixed with a liquid carrier to form a concentrate suitable for subsequent mixing with a liquid carrier. The application of the composition to the plant or its locus in solid or concentrate form especially where water is present in the vicinity of the plant through natural precipitation is also within the ambit of the invention.

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- In water, suitably the composition is diluted for use to a level of 10 to 500 mg/l and preferably 20 to 300mg/l. The dilution is suitably selected according to the composition used, the type of application, the state of growth of the plants to be treated and other factors known to those skilled in the art.

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This invention relates to a process for producing a composition and to a method of treating plants, for example killing or controlling weeds by applying a reduced amount of the active ingredient(s), suitably diluted in water, than that normally recommended for such active(s) against such weeds. In addition the invention allows for the avoidance of subsequent applications of the said actives, thus further reducing the amount of pesticide used.

The invention is illustrated by the following non-limiting examples.

10 Examples 1 to 8 and Comparative Examples A to C

A series of compositions produced according to the invention having the formulations set out in Table 1 were prepared by blending the active which had been previously milled and GALORYL DT505, a naphthalene sulfonic acid, until uniform and then air-milling using a Gem-T airmill. Sodium carbonate and filler were added to this premix and blended in a high speed mixer until uniform. 12% by weight based on the mixture of distilled water was added whilst blending. The wetted pre-mix which was a free flowing powder was fed to a basket extruder as described in EP-A-812256 through a 1mm screen. A compacted extrudate was obtained and the resulting granules dried at 60°C for 8 minutes. A paste was not formed prior to extrusion. The dried granules were then sieved through 2mm and 500micron sieves.

The Assay determinations were carried out using a Hewlett Packard 1050 HPLC machine. HPLC Method LC29 was used for the analysis. An analytical standard purchased from QMx was used as the reference material.

Example 3 with half lactose and half clay filler was extruded more readily than the Examples 1 and 2.

30 Table 1

| Example | Bensulfuron-Methyl Assay (%) | Sodium Carbonate (%) | Galoryl DT505 (%) | Filler (balance %) |
|---------|------------------------------|----------------------|-------------------|--------------------|
| 1       | 5                            | 5                    | 10                | Lactose            |
| 2       | 5                            | 2                    | 10                | Lactose            |

|   |    |             |      |                               |
|---|----|-------------|------|-------------------------------|
| 3 | 5  | 3           | 10   | Lactose/China Clay<br>(50/50) |
| 4 | 60 | 10          | 20   | Lactose                       |
| 5 | 60 | 15          | 10   | Lactose                       |
| 6 | 60 | 20          | 10   | Lactose                       |
| 7 | 60 | 20          | 12.5 | Lactose                       |
| 8 | 60 | 20 (milled) | 12.5 | Lactose                       |

Various physical characteristics of the composition were ascertained using the following methods and the results are set out in Table 2 below.

#### 5 Suspensibility

0.5g of product, weighed to four decimal places, was added to 100ml tap water at 20°C to the 100ml mark of a 100ml measuring cylinder. The cylinder top was placed on the cylinder and then inverted through 180° and back about the centre of the cylinder 30 times. The rate of inversion was one inversion per second. If, after 30  
10 inversions not all of the product has dispersed, further inversions were carried out in groups of 5 until all of the product had dispersed, or until 60 inversions had been completed.

The cylinder was left to stand undisturbed for 30 minutes. After this time 10ml of the  
15 dispersion was removed from the 50ml mark on the cylinder pipette and placed in a pre-weighed and dried evaporating basin. The water was evaporated using a water bath. When all the water has evaporated the basin was further dried by microwaving for 3 minutes on medium power. The basin was then re-weighed when it had cooled to room temperature.

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The suspensibility is calculated using the formula below;

$$\% \text{ Suspensibility} = \frac{(\text{Weight of residue/g} - Y^*) \times 1000}{\text{Weight of product/g}}$$

25  $Y^*$  = mass of residue from 10ml tap water at time of measurement

#### Combined Wet Sieve Residue Test for 75µm and 150µm Sieves

5g of product, weighed to four decimal places was added to a 250ml beaker containing 100ml tap water. The product was stirred manually, until the suspension is uniform or for 5 minutes maximum.

- 5 A 75µm sieve and a 150µm sieve were pre-wetted under a tap. The 150µm sieve was placed on top of the 75µm sieve and the suspension was poured through both sieves and any residue washed for up to 2 minutes with tap water flowing at a rate of 3 litres per minute. The beaker was thoroughly rinsed to ensure that all the contents were transferred to the sieves.

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The sieves were separated and the residue on each sieve transferred to a pre-dried and pre-weighed evaporating basin using deionised water in a wash bottle. The water was evaporated using a water bath. When all the water has evaporated the basin was further dried by microwaving for 3 minutes on medium power. The basin

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was then re-weighed when it had cooled to room temperature.

The 150µm wet sieve residue is calculated using the formula below:

$$\% \text{ Residue} = \frac{\text{Weight of residue/g} \times 100}{\text{Weight of product/g}}$$

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The 75µm wet sieve residue is calculated using the formula below:

$$\% \text{ Residue} = \frac{(\text{weight of residue/g} + \text{weight of residue from 150µm sieve/g}) \times 100}{\text{weight of product/g}}$$

## 25 Dispersibility

100ml tap water was poured into a 100ml measuring cylinder. 1.0g product was quickly added, and the cylinder top placed on the cylinder before inverting through 180° and back 10 times about an axis through the centre of the cylinder. If after 10 inversions not all of the product had dispersed the inversions were continued in lots

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of 5 until all of the product has dispersed, or until 60 inversions was reached.

Dispersibility Rating Star System:

- \* - Very few or no granule bits / specks

- \*\* - A few granule bits / specks
- \*\*\* - Lots of bits / specks
- \*\*\*\* - Less than 5 undispersed granules
- \*\*\*\*\* - More than 5 undispersed granules

5

Table 2

| Example | Dispersibility<br>(Number of<br>inversions) | Suspensibility<br>(%) | Wet<br>Sieve<br>75micron | Wet Sieve<br>150micron | Moisture<br>(%) | Assay<br>(%) |
|---------|---|-----------------------|--------------------------|------------------------|-----------------|--------------|
| 1       | 25*   | 97                    | 0.01                     | 0.01                   | 0.67            | 5.02         |
| 2       | 25 no stars                                 | 100                   | 0.03                     | 0.06                   | 0.80            | 5.18         |
| 3       | 25*   | 94                    | 0.01                     | 0.03                   | 1.00            | 5.20         |
| 4       | 40**  | 97                    | 0.03                     | 0.08                   | 1.20            | 59.73        |
| 5       | 50**  | 100                   | 0.01                     | 0.01                   | 1.09            | 60.35        |
| 6       | 50**  | 95                    | 0.02                     | 0.03                   | 1.51            | 60.03        |
| 7       | 60**  | 96                    | 0.06                     | 0.08                   | 0.89            | 59.34        |
| 8       | 60*** / 100**                               | 97                    | 0.10                     | 0.14                   | 0.60            | 56.25        |

10 Examples 3 and 8 were subjected to a stability test in which the compositions were stored in a sealed container for 2 weeks at the specified temperature. The results are set out in Table 3.



Table 3

|           |                       | RT    | 37°C  | 54°C  | 25/40°C | F/T   | 5°C   |
|-----------|-----------------------|-------|-------|-------|---------|-------|-------|
| Example 3 | Suspensibility (%)    | 88    | 90    | 92    | 90      | 89    | 90    |
|           | Wet Sieve (150um) (%) | 0.01  | 0.01  | 0.01  | 0.01    | 0.01  | 0.01  |
|           | Wet Sieve (75um) (%)  | 0.01  | 0.02  | 0.01  | 0.04    | 0.02  | 0.02  |
|           | Dispersibility        | 25*   | 25*   | 25*   | 25*     | 25*   | 25*   |
|           | Moisture (%)          | 1.31  | 0.98  | 1.20  | 1.00    | 1.12  | 1.02  |
|           | Assay (%)             | 5.13  | 5.18  | 5.12  | 5.14    | 5.21  | 5.09  |
|           | Active remaining (%)  | 98.7  | 99.6  | 98.5  | 98.8    | 100.2 | 97.9  |
| Example 8 | Suspensibility (%)    | 95    | 93    | 96    | 96      | 98    | 97    |
|           | Wet Sieve (150um) (%) | 0.02  | 0.01  | 0.01  | 0.01    | 0.01  | 0.01  |
|           | Wet Sieve (75um) (%)  | 0.03  | 0.01  | 0.01  | 0.01    | 0.02  | 0.01  |
|           | Dispersibility        | 100*  | 100*  | 100*  | 100*    | 100*  | 100*  |
|           | Moisture (%)          | 1.80  | 1.22  | 1.89  | 1.21    | 2.03  | 1.49  |
|           | Assay (%)             | 55.91 | 55.96 | 56.13 | 56.04   | 55.95 | 55.59 |
|           | Active remaining (%)  | 99.4  | 99.5  | 99.8  | 99.6    | 99.5  | 98.8  |

These results demonstrate that the compositions maintained their stability to an acceptable level for a period of two weeks.

#### Solubility Data

- 5 The solubility characteristics of compositions according to the invention were tested. Three known compositions were also tested for comparative purposes. The three comparative compositions were as follows:

Comparative Example A:

LONDAX 60 – a commercially available bensulfuron-methyl product having 60% bensulfuron-methyl, available from Du Pont.

Comparative Example B:

| Component                            | Trade Name     | %w/w |
|--------------------------------------|----------------|------|
| Bensulfuron-methyl                   |                | 64   |
| Naphthalene sulfonic acid            | GALOROYL DT505 | 13   |
| Formaldehyde condensate, sodium salt | GALOROYL MT704 | 1    |
| Lactose                              | Lactose        | 22   |

5

Comparative Example C:

| Component                            | Trade Name     | %w/w |
|--------------------------------------|----------------|------|
| Bensulfuron-methyl                   |                | 5    |
| Naphthalene sulfonic acid            | GALOROYL DT505 | 30   |
| Formaldehyde condensate, sodium salt | GALOROYL MT704 | 1    |
| China Clay                           | China Clay GTY | 64   |

Comparative Examples B and C were prepared by the same method as the compositions of the invention.

10

The solubility test method was carried out in a 1 litre jacketed vessel, cooled by a chiller to 5°C (propan-1,2-diol/water) and was agitated using a magnetic stirrer, stirring at maximum speed. 1 litre of CIPAC D water was measured into the jacketed vessel and left stirring until thermal equilibrium had been reached. The pH of the water was measured and recorded (6.5 for CIPAC D water). The composition to be tested was then added, under continued stirring, and the stopwatch started. Aliquots of 2ml were taken after 5, 15, 30 and 60 minutes, using a syringe, filtered immediately through a Whatman GF/A filter paper and the filtrate analysed for dissolved bensulfuron-methyl.

20

### Results

The results of the solubility tests are set out in Tables 4 to 6.

Table 4

| Test Sample           | Application Rate<br>g/hectare | Bensulfuron Applied<br>g/hectare | % Bensulfuron dissolved |       |       |       | Bensulfuron Dissolved<br>g/hectare |
|-----------------------|-------------------------------|----------------------------------|-------------------------|-------|-------|-------|------------------------------------|
|                       |                               |                                  | 5min                    | 15min | 30min | 60min |                                    |
| Comparative Example A | 100                           | 60.0                             | 3.2                     | 3.4   | 3.5   | 3.9   | 2.3                                |
| Comparative Example B | 100                           | 60.0                             | 14.9                    | 15.5  | 16.3  | 15.0  | 9.0                                |
| Comparative Example C | 300                           | 15.0                             | 63.3                    | 63.1  | 62.7  | 63.2  | 9.5                                |

Table 5

| Example | Application Rate<br>g/hectare | Bensulfuron Applied<br>g/hectare | % Bensulfuron dissolved |       |       |       | Bensulfuron Dissolved<br>g/hectare |
|---------|-------------------------------|----------------------------------|-------------------------|-------|-------|-------|------------------------------------|
|         |                               |                                  | 5min                    | 15min | 30min | 60min |                                    |
| 1       | 300                           | 15.0                             | 97.1                    | 96.7  | 96.9  | 97.4  | 14.6                               |
| 2       | 300                           | 15.0                             | 75.8                    | 88.0  | 90.2  | 93.0  | 14.0                               |
| 3       | 300                           | 15.0                             | 70.1                    | 96.8  | 98.3  | 99.0  | 14.9                               |
| 4       | 100                           | 60                               | 41.2                    | 50.5  | 52.1  | 52.9  | 31.7                               |
| 5       | 100                           | 60                               | 57.6                    | 74.3  | 78.4  | 79.7  | 47.8                               |
| 6       | 100                           | 60                               | 67.9                    | 85.8  | 89.5  | 91.5  | 54.9                               |
| 7       | 100                           | 60                               | 66.2                    | 86.8  | 90.8  | 92.6  | 55.6                               |
| 8       | 100                           | 60                               | 65.3                    | 96.3  | 98.2  | 97.8  | 58.7                               |

5. A further solubility test was carried out on the wetted premix used in the preparation of the composition of Example 8, that is the formulation prior to extrusion. The results are shown below.

Table 6

|                     |     |    |      |      |      |      |      |
|---------------------|-----|----|------|------|------|------|------|
| Premix of Example 8 | 100 | 60 | 83.8 | 90.6 | 93.6 | 96.5 | 57.9 |
|---------------------|-----|----|------|------|------|------|------|

10

Table 7 below shows the change in pH in each test solution, after 60 minutes solubility testing.

Table 7

| Example | Bensulfuron<br>Assay (%) | Sodium<br>Carbonate<br>(%) | Application<br>Rate<br>g/hectare | Initial pH<br>(CIPAC D<br>Water at 5°C) | Final pH<br>(CIPAC D<br>water at 5°C) |
|---------|--------------------------|----------------------------|----------------------------------|---|---------------------------------------|
| 1       | 5                        | 5                          | 300                              | 6.60                                    | 9.55                                  |
| 2       | 5                        | 2                          | 300                              | 6.40                                    | 8.50                                  |
| 3       | 5                        | 3                          | 300                              | 6.40                                    | 9.10                                  |
| 4       | 60                       | 10                         | 100                              | 6.40                                    | 8.60                                  |
| 5       | 60                       | 15                         | 100                              | 6.70                                    | 8.80                                  |
| 6       | 60                       | 20                         | 100                              | 6.50                                    | 8.95                                  |
| 7       | 60                       | 20                         | 100                              | 6.40                                    | 9.00                                  |
| 8       | 60                       | 20 (milled)                | 100                              | 6.60                                    | 9.00                                  |

## CLAIMS

1. A process for the production of a granular composition comprising an agrochemical active agent, a basic, inorganic carbonate salt and optionally a dispersing agent wherein a 1% aqueous solution of the composition has a pH of at least 8.0 when subjected to CIPAC Test MT75.3 which process comprises forming a premix in the form of a free-flowing powder comprising the active, the basic inorganic salt and optionally a dispersing agent surfactant with at least one of the components of the pre-mix being liquid, without forming a paste, extruding the pre-mix to form the granules and optionally drying the granules.
2. A process according to claim 1 in which the basic, inorganic salt in the composition is such that a 1% solution of the composition provides a pH of at least 8.5.
3. A process according to claim 1 or claim 2 in which the active comprises a low use rate agrochemical active.
4. A process according to claim 3 in which the low use rate active comprises a sulfonyl urea.
5. A process according to claim 4 in which the low use rate active is selected from bensulfuron-methyl and chlorsulfuron.
6. A process according to any one of claims 3 to 5 which comprises at least 50% active by weight.
7. A process according to claim 6 in which at least 25% of the active agent in the composition is dissolved after 15 minutes at 5°C.
8. A process according to claim 7 in which at least 40% of the active agent in the composition is dissolved after 5 minutes at 5°C.
9. A process according to any one of claims 3 to 5 which comprises less than 7 % active by weight.

10. A process according to claim 9 in which at least 70% of the active agent in the composition is dissolved in water after 15 minutes at 5°C.
- 5 11. A process according to claim 10 in which at least 70% of the active agent in the composition is dissolved after 5 minutes at 5°C.
12. A process according to any one of the preceding claims in which the basic, inorganic carbonate salt comprises an alkali metal salt.
- 10 13. A process according to claim 12 in which the alkali metal salt comprises sodium carbonate.
- 15 14. A process according to any one of the preceding claims in which the basic, inorganic carbonate salt is anhydrous.
- 15 15. A process according to any one of the preceding claims in which the active agrochemical is selected from bensulfuron-methyl and chlorsulfuron and the basic inorganic salt comprises sodium carbonate.
- 20 16. A process according to claim 15 in which the actives comprise i) bensulfuron-methyl and propanil or ii) chlorsulfuron and isoproturon.
- 25 17. A process according to any one of the preceding claims in which the weight ratio of total agrochemical active in the composition to basic inorganic salt is not more than 10:1 and is at least 1:3.
- 30 18. A process according to any one of the preceding claims which further comprises a dispersing agent selected from a non-ionic surfactant and an anionic surfactant.
- 35 19. A process according to claim 18 selected from a non-ionic surfactant which has a Zeta Potential of about 0mV and an anionic surfactant which has a Zeta Potential in excess of -30mV.

20. A method of increasing the rate and degree of dissolution of an agrochemical active in an aqueous medium which method comprises formulating a composition comprising the agrochemical active with a basic, inorganic salt such that a 1% aqueous solution of the composition has a pH of at least 8 when subjected to CIPAC Test MT75.3 and producing the composition by extruding a premix comprising the active and the salt and optionally a dispersing agent wherein a paste is not formed prior to extrusion.
21. Use of a basic, inorganic salt in an agrochemical composition as defined in any one of claims 1 to 19, to increase the proportion of the active in the composition that is dissolved when mixed with aqueous solution and thereby to improve delivery of the active to a plant or a locus to be treated.
22. Use according to claim 21 in which the composition comprises an active agent at a level up to 10% by weight of the composition, and at least 70% of the active agent in the composition is dissolved in CIPAC D water at 5°C after 15 minutes.
23. Use according to claim 21 in which the composition comprises an active agent at a level more than 10% by weight of the composition, and at least 25% of the active agent in the composition is dissolved in CIPAC D water at 5°C after 15 minutes.
24. A composition obtainable by a process according to any one of claims 1 to 19 and wherein the composition contains an active agent at a level up to 10% by weight of the composition, and the proportion of active dissolved after 15 minutes at 5°C is at least 30% and more than the proportion of active dissolved in a comparative composition in which the salt is replaced by an inert filler.
25. A composition obtainable by a process according to any one of claims 1 to 19 and wherein the composition contains an active agent at a level of at least 50% by weight of the composition, and the proportion of active dissolved after 15 minutes at 5°C is at least 100% and more than the proportion of active dissolved in a comparative composition in which the salt is replaced by an inert filler.

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(57) Abstract: A water dispersible granular composition containing an agrochemical active agent, particularly a low use-rate active, and a particular basic, inorganic salt wherein a 1% aqueous solution of the composition has a pH of at least 8.0, and which is made by extruding a premix in the form of a free-flowing powder and without forming a paste provides improved bioavailability of the active. The invention is particularly useful in improving delivery of active from compositions having mixed actives and low levels of sulphonamide active.

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| C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT |  |                       |
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